EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L6	1	resin.clm. and (beta-elimination or "beta elimination").clm. and glycopeptide	US-PGPUB	OR	ON	2006/10/20 16:05
L7	5	resin.clm. and (beta-elimination or "beta elimination").clm.	US-PGPUB	OR	ON	2006/10/20 16:05

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FILE 'CAPLUS' ENTERED AT 15:44:44 ON 20 OCT 2006 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.

PLEASE SEE "HELP USAGETERMS" FOR DETAILS. COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS) FILE 'EMBASE' ENTERED AT 15:44:44 ON 20 OCT 2006 Copyright (c) 2006 Elsevier B.V. All rights reserved. FILE 'BIOSIS' ENTERED AT 15:44:44 ON 20 OCT 2006 Copyright (c) 2006 The Thomson Corporation => beta-elimination or "beta elimination" 7586 BETA-ELIMINATION OR "BETA ELIMINATION" => resin or "solid phase" or "on-resin" 1032838 RESIN OR "SOLID PHASE" OR "ON-RESIN" => glycopeptides or "o-glcnac" 24823 GLYCOPEPTIDES OR "O-GLCNAC" => 11 and 12 and 13 22 L1 AND L2 AND L3 => dup rem 14 PROCESSING COMPLETED FOR L4 9 DUP REM L4 (13 DUPLICATES REMOVED) => d ibib abs total 15 ANSWER 1 OF 9 MEDLINE on STN DUPLICATE 1 ACCESSION NUMBER: 2001383288 MEDLINE DOCUMENT NUMBER: PubMed ID: 11325260 TITLE: Use of fluorobenzoyl protective groups in synthesis of glycopeptides: beta-elimination of O-linked carbohydrates is suppressed. AUTHOR: Sjolin P; Kihlberg J CORPORATE SOURCE: Organic Chemistry, Department of Chemistry, Umea University, SE--901 87 Umea, Sweden. SOURCE: The Journal of organic chemistry, (2001 May 4) Vol. 66, No. 9, pp. 2957-65. Journal code: 2985193R. ISSN: 0022-3263. PUB. COUNTRY: United States DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE) LANGUAGE: English FILE SEGMENT: Priority Journals ENTRY MONTH: 200107 ENTRY DATE: Entered STN: 9 Jul 2001 Last Updated on STN: 9 Jul 2001 Entered Medline: 5 Jul 2001 AΒ Fluorobenzoyl groups have been investigated as alternatives to acetyl and benzoyl protective groups in carbohydrate and glycopeptide synthesis. D-Glucose and lactose were protected with different fluorobenzoyl groups and then converted into glycosyl bromides in high yields (>80% over two steps). Glycosylation of protected derivatives of serine with these donors gave 1,2-trans glycosides in good yields (approximately 60--70%) and excellent stereoselectivity without formation of ortho esters. The resulting glycosylated amino acid building blocks were then used in solid-phase synthesis of two model O-linked glycopeptides known to be unusually sensitive to betaelimination on base-catalyzed deacylation. When either a 3-fluoro- or a 2,5-difluorobenzoyl group was used for protection of each of the two model glycopeptides the extent of betaelimination decreased from 80% to 10% and from 50% to 0%, respectively, as compared to when using the ordinary benzoyl group. Fluorobenzoyl groups thus combine the advantages of the benzoyl group in

formation of glycosidic bonds (i.e., high stereoselectivity and low levels of ortho ester formation) with the ease of removal characteristic of the

acetyl group.

AUTHOR:

L5 ANSWER 2 OF 9 MEDLINE on STN DUPLICATE 2

ACCESSION NUMBER: 2001138871 MEDLINE DOCUMENT NUMBER: PubMed ID: 11192238

TITLE: Synthesis of tumor associated sialyl-T-

glycopeptides and their immunogenicity. Komba S; Werdelin O; Jensen T; Meldal M

CORPORATE SOURCE: Carlsberg Laboratory, Department of Chemistry, Valby,

Copenhagen, Denmark.

SOURCE: Journal of peptide science: an official publication of the

European Peptide Society, (2000 Dec) Vol. 6, No. 12, pp.

585-93.

Journal code: 9506309. ISSN: 1075-2617.

PUB. COUNTRY: England: United Kingdom

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 200103

ENTRY DATE: Entered STN: 4 Apr 2001

Last Updated on STN: 4 Apr 2001 Entered Medline: 8 Mar 2001

AB Sialyl-T-glycopeptides were synthesized by solidphase techniques, using a PEGA resin as the solid

support. An appropriately protected building block containing

alpha-Neu5Ac-(2 --> 3)-beta-Gal-(1 --> 3)-alpha-GalN3-(1-->) attached to

Fmoc-Thr/Ser-OPfp was employed in a solid phase

glycopeptide assembly of a 10-mer glycopeptide, using a general Fmoc/OPfp-ester strategy. Reduction of the azido group of the GalN3 residue was effected on solid-phase, using DTT and

DBU. After acidolytic cleavage from the resin, the methyl ester of the sialic acid residue and acetyl groups were removed with 30%

NaOMe/MeOH in MeOH and water pH 14, at -30 degrees C for 2 h. At this low temperature, the highly basic conditions did not result in any detectable beta-elimination. However, one O-acetyl group, located

at the 2-position of the Gal was resistant to hydrolysis. To remove this remaining acetyl group, reaction with hydrazine hydrate in CHCl3 and MeOH at room temperature for 2.5 h was successful. The two target sequences of

sialyl-T-glycopeptides were obtained in good yield. In contrast to the the analogs carrying the T-antigen, the Sial-T-

glycopeptides were nonimmunogenic, supporting the idea that the

sialylation is a method of circumventing the recognition by the immune system.

L5 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1999:357920 CAPLUS

DOCUMENT NUMBER: 131:214543

TITLE: Synthesis of deoxy and alanine-substituted derivatives

of a T cell stimulating glycopeptide - An

investigation of conditions for cleavage from the

solid phase and deprotection

AUTHOR(S): Sjolin, Petter; George, Shaji K.; Bergquist,

Karl-Erik; Roy, Sarbari; Svensson, Anette; Kihlberg,

Jan

CORPORATE SOURCE: Department of Chemistry, Organic Chemistry, Ume

University, Ume, SE-901 87, Swed.

SOURCE: Journal of the Chemical Society, Perkin Transactions

1: Organic and Bio-Organic Chemistry (1999), (12),

1731-1742

CODEN: JCPRB4; ISSN: 0300-922X

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 131:214543

GΙ

$$\begin{array}{c} \text{HO} \\ \text{OH} \\$$

Little is known concerning how T cells recognize glycopeptides AB presented by MHC mols. on antigen-presenting cells. In order to probe the specificity of helper T cells elicited on immunization of mice with neoglycopeptide I (X = -Gln-Ile-Asn-Ser-Arg61-NH2, Y = H-Asp52-Tyr-Gly-Ile-) which has the disaccharide galabiose [Gal α (1 4) Gal β] O-linked to serine 56 in the hen egg lysozyme peptide HEL(52-61), we have prepared three sets of glycopeptides. These are: the 6- and 6'-deoxygalabiose analogs of I, two glycopeptides in which the galabiose moiety of I has been replaced by galactose and lactose, resp., and an alanine-scan series of I in which all amino acid residues, apart from 54 and 56, were replaced by alanine, one by one. deoxygenated galabiose donors, activated either as an anomeric trichloroacetimidate or as a β -acetate, were used for glycosylation of Fmoc-Ser-OPfp. The resulting, and other, glycosylated amino acids were then used as building blocks in solid-phase synthesis of the target glycopeptides. It was found that improved yields of glycopeptides could be obtained if cleavage from the solid phase was performed at 40 °C instead of at room temperature In the final base-catalyzed deprotection of the carbohydrate moiety, removal of O-benzoyl groups was accompanied by substantial . beta.-elimination. For one of the glycopeptides even deacetylation required carefully controlled conditions in order to avoid β -elimination. The glycopeptides described in the present work have been evaluated for binding to I-Ak class II MHC mols., as well as for their ability to stimulate helper T cell hybridomas obtained from mice immunized with the galabiosylated glycopeptide I (no data).

REFERENCE COUNT:

THERE ARE 57 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2006 ACS on STN

57

ACCESSION NUMBER:

2000:859763 CAPLUS

DOCUMENT NUMBER:

134:127543

TITLE:

Glycobiology of MSP-1 and MSP-2: potential malaria

vaccine candidate glycoproteins

AUTHOR(S):

Nasir-Ud-Din; Hoessli, Daniel C.; Khan, Abbas H.

CORPORATE SOURCE:

Institute of Biochemistry, University of Balochistan,

Quetta, Pak.

SOURCE:

Journal of the Chemical Society of Pakistan (1999),

21(3), 299-304

CODEN: JCSPDF; ISSN: 0253-5106

PUBLISHER: Chemical Society of Pakistan

DOCUMENT TYPE: Journal LANGUAGE: English

Metabolic labeling of Plasmodium falciparum parasites with [3H]GlcN, AB

[3H]Man, [3H]Gal and [3H]ethanolamine, and subsequent purification by SDS-PAGE of the labeled material provided effective labeling of the MSP-1 195 kDa,

and MSP-2, 42-53 kDa, glycoproteins. Reductive β -

elimination of the MSP-2 released from the gel consisted of

glycopeptides containing labeled sugars. Processing of the eliminated components and identification of the sugar residues demonstrated the presence of N-acetylglucosaminitol and N-acetylgalactosaminitol amongst

other labeled sugars. Reductive β -elimination

with sodium hydroxide-sodium borotritideborohydride showed the presence of glucosaminitol and alanine in the hydrolysis products. The MSP-2 was

retained on solid phase wheat-germ agglutinin and was

released from the lectin by treatment with GlcNAc. Upon treatment with O-glycanase the MSP-2 glycoprotein released labeled amino sugar, and derived oligosaccharides on treatment with exoglycosidases released

labeled components corresponding to the metabolically incorporated sugars. Labeled Gal was incorporated into the MSP-2 glycoprotein using [3H]UDP-Gal and galactosyltransferase. The galactosylated glycoprotein released labeled Gal upon treatment with β -galactosidase. The results of the

present study suggest that the carbohydrate chains of the MSP-2 glycoprotein are attached to the protein backbone via GlcNAc- and GalNAc-serine/threonine in O-glycosyl linkage and the glycoprotein has terminal GlcNAc and Gal residues. The carbohydrate moieties of MSP-2,

glycoprotein consist mainly of short chains linked to the protein core. Mannosamine inhibits biosynthesis as well as parasitemia of P. falciparum. REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5ANSWER 5 OF 9 MEDLINE on STN DUPLICATE 3

ACCESSION NUMBER: 1998013338 MEDLINE DOCUMENT NUMBER: PubMed ID: 9352084

TITLE: Carbohydrate moiety of Plasmodium falciparum glycoproteins:

the nature of the carbohydrate-peptide linkage in the MSP-2

glycoprotein.

AUTHOR: Khan A H; Qazi A M; Hoessli D C; Torred-Duarte A P; Senaldi

G; Qazi M H; Walker-Nasir E; Nasir-ud-Din

CORPORATE SOURCE: Institute of Biochemistry, University of Balochistan,

Quetta, Pakistan.

SOURCE: Biochemistry and molecular biology international, (1997

Oct) Vol. 43, No. 3, pp. 655-68.

Journal code: 9306673. ISSN: 1039-9712.

PUB. COUNTRY: Australia

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 199712

ENTRY DATE: Entered STN: 9 Jan 1998

Last Updated on STN: 9 Jan 1998 Entered Medline: 11 Dec 1997

AΒ Metabolic labelling of Plasmodium falciparum parasites with [3H]GlcN, [3H]Man, [3H]Gal and [3H]ethanolamine, and subsequent purification by SDS-PAGE of the labelled material provided effective labelling of the MSP-1, 195 kDa, and MSP-2, 42-53 kDa, glycoproteins. Reductive

beta-elimination of the MSP-2 released from the gel consisted of glycopeptides containing labelled sugars.

Processing of the eliminated components and identification of the sugar residues demonstrated the presence of N-acetylglucosaminitol and N-acetylgalactosaminitol amongst other labelled sugars. Reductive

beta-elimination with sodium hydroxide-sodium

borotritide-borohydride showed the presence of glucosaminitol and alanine in the hydrolysis products. The MSP-2 was retained on solid phase wheat-germ agglutinin and was released from the lectin by

treatment with GlcNAc. Upon treatment with O-glycanase the MSP-2 glycoprotein released labelled amino sugar, and derived oligosaccharides on treatment with exoglycosidases released labelled components corresponding to the metabolically incorporated sugars. Labelled Gal was incorporated into the MSP-2 glycoprotein using [3H]UDP-Gal and galactosyltransferase. The galactosylated glycoprotein released labelled Gal upon treatment with beta-galactosidase. The results of the present study suggest that the carbohydrate chains of the MSP-2 glycoprotein are attached to the protein backbone via GlcNAc- and GalNAc-serine/threonine in O-glycosyl linkage and the glycoprotein has terminal GlcNAc and Gal residues. The carbohydrate moieties of MSP-2, glycoprotein consist mainly of short chains linked to the protein core.

ANSWER 6 OF 9 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 4 L5

ACCESSION NUMBER:

1996:355015 CAPLUS

DOCUMENT NUMBER:

125:143268

TITLE:

Piperidine is preferred to morpholine for Fmoc

cleavage in solid phase

glycopeptide synthesis as exemplified by preparation

of glycopeptides related to HIV gp120 and

AUTHOR(S):

SOURCE:

Vuljanic, Tatjana; Bergquist, Karl-Erik; Clausen,

Henrik; Roy, Sarbari; Kihlberg, Jan

CORPORATE SOURCE:

Org. Chem. 2, Cent. Chem. Chem. Eng., Lund Inst.

Technol., Lund Univ., Lund, S-221 00, Swed.

Tetrahedron (1996), 52(23), 7983-8000

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER:

Elsevier Journal

DOCUMENT TYPE: LANGUAGE:

English

OTHER SOURCE(S):

CASREACT 125:143268

Protected derivs. of the Tn antigens Fmoc-Ser/Thr(Ac3GalNAca)-OH (I; Fmoc = 9-fluorenylmethoxycarbonyl) were prepared by glycosylation of Fmoc-Ser/Thr-OCH2CH:CH2 with 3,4,6-tri-O-acetyl-2-azido-2-deoxy-Dgalactopyranosyl chloride, followed by conversion of the azido group to an acetamide and deallylation. Building blocks I were used for solid phase synthesis of glycopeptides related to HIV gp 120 and mucins. In these syntheses, piperidine was found to give efficient Fmoc removal whereas deprotection with morpholine was slow and incomplete for some steps. In contrast to previous concerns, $\boldsymbol{\beta}$ elimination and epimerization of glycopeptide stereocenters was not encountered when piperidine was used for Fmoc deprotection. it was found that for Cys-containing glycopeptides, de-O-acetylation with methanolic ammonia had to be performed before side chain deprotection and cleavage from the solid phase.

ANSWER 7 OF 9

MEDLINE on STN

DUPLICATE 5

ACCESSION NUMBER: DOCUMENT NUMBER:

95013041 MEDLINE PubMed ID: 7928083

TITLE:

Susceptibility of glycans to beta-

AUTHOR:

elimination in Fmoc-based O-glycopeptide synthesis. Meldal M; Bielfeldt T; Peters S; Jensen K J; Paulsen H;

Bock K

CORPORATE SOURCE:

Carlsberg Laboratory, Department of Chemistry, Valby,

SOURCE:

Copenhagen, Denmark. International journal of peptide and protein research,

(1994 Jun) Vol. 43, No. 6, pp. 529-36. Journal code: 0330420. ISSN: 0367-8377.

PUB. COUNTRY:

Denmark

DOCUMENT TYPE:

Journal; Article; (JOURNAL ARTICLE)

LANGUAGE:

English

FILE SEGMENT:

Priority Journals

ENTRY MONTH:

199411

ENTRY DATE:

Entered STN: 22 Dec 1994

Last Updated on STN: 5 Aug 1996

Entered Medline: 1 Nov 1994

ΔR In order to investigate the possible extent of betaelimination occurring in Fmoc-based continuous-flow solid -phase glycopeptide synthesis, the influence of the pKb of the base used for N alpha-deprotection has been studied. A glycosylated pentapeptide was synthesized using 50% morpholine, 10% piperidine or 2% DBU, respectively, in DMF for deprotection. The dehydropentapeptide N alpha-Ac-Thr-Thr-delta Aba-Val-Thr-NH2, which would be formed in the case of beta-elimination, was prepared independently and used as a control in HPLC analysis; however, this product was not formed under any of the deprotection conditions applied. Furthermore, a 23 amino acid long glycopeptide from human intestinal mucin was prepared using 2% DBU as a base for Fmoc cleavage, and similarly no betaelimination was observed. The glycopeptide products were subjected to a prolonged treatment with sodium hydroxide in methanol/water without significant formation of byproducts, and the pure glycopeptides were isolated and characterized by 1H-NMR spectroscopy.

ANSWER 8 OF 9 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 6 Ľ5

ACCESSION NUMBER:

1994:135109 CAPLUS

DOCUMENT NUMBER:

120:135109

TITLE:

Piperidine is preferable to morpholine for Fmoc

cleavage in solid phase synthesis

of O-linked glycopeptides

AUTHOR(S):

Kihlberg, Jan; Vuljanic, Tatjana

CORPORATE SOURCE:

Org. Chem. 2, Chem. Cent., Lund Inst. Technol., Lund,

S-221 00, Swed.

SOURCE:

Tetrahedron Letters (1993), 34(38), 6135-8

CODEN: TELEAY; ISSN: 0040-4039

DOCUMENT TYPE:

Journal

LANGUAGE:

English

Morpholine gives slow and incomplete 9-fluorenylmethoxycarbonyl (Fmoc) removal in a solid-phase synthesis of and O-linked

HIV-related glycopeptide, resulting in substantial byproduct formation. significant improvement was obtained by replacement of morpholine with piperidine, and, in contrast to common belief, β -

elimination was not observed

·L5 ANSWER 9 OF 9 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER:

1989:132358 BIOSIS

DOCUMENT NUMBER:

PREV198987067011; BA87:67011

TITLE:

SOLID-PHASE SYNTHESIS OF O GLYCOPEPTIDE

SEOUENCES.

AUTHOR(S):

PAULSEN H [Reprint author]; MERZ G; WEICHERT U

CORPORATE SOURCE:

INST ORGANISCHE CHEM, UNIV MARTIN-LUTHER-KING-PLATZ 6,

D-2000 HAMBURG 13

SOURCE:

Angewandte Chemie International Edition in English, (1988)

Vol. 27, No. 10, pp. 1365-1367. CODEN: ACIEAY. ISSN: 0570-0833.

DOCUMENT TYPE:

Article

FILE SEGMENT: LANGUAGE:

BA ENGLISH

ENTRY DATE:

Entered STN: 10 Mar 1989

Last Updated on STN: 10 Mar 1989

AB 4-Alkoxybenzyl alcohol resins are suitable supports for the solid-phase synthesis of O-glycopeptides.

This was shown by the syntheses of the hexapeptides 1 and 2 in 55 and 48% yield, respectively, based on the first amino acid to be coupled. .

beta.-Elimination, racemization, or glycoside cleavage

were not observed.

=> glycopeptide (3a) (separation or purification) and review 1.6 6 GLYCOPEPTIDE (3A) (SEPARATION OR PURIFICATION) AND REVIEW => dup rem 16

PROCESSING COMPLETED FOR L6

L7 4 DUP REM L6 (2 DUPLICATES REMOVED)

=> d ibib abs total

L7 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2002:866421 CAPLUS

DOCUMENT NUMBER: 139:138830

TITLE: Glycopeptide Antibiotics-bonded Chiral Stationary

Phases for Chiral Separation in HPLC

AUTHOR(S): Ye, Xiaoxia; Yu, Xiong

CORPORATE SOURCE: Shanghai institute of Pharmaceutical Industry,

Shanghai, 200040, Peop. Rep. China

SOURCE: Zhongguo Yiyao Gongye Zazhi (2002), 33(4), 194-198

CODEN: ZYGZEA; ISSN: 1001-8255

PUBLISHER: Zhongguo Yiyao Gongye Zazhi Bianjibu

DOCUMENT TYPE: Journal; General Review

LANGUAGE: Chinese

AB A review 25 refs. with on glycopeptide antibiotics-bonded chiral

stationary phases for chiral separation in HPLC with subdivision headings: (1) structures of glycopeptide antibiotics; mechanism of chiral recognition; (3) working modes; (4) principle of complementary separation; (5) universal

property; and performance of column.

L7 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2001:121476 CAPLUS

DOCUMENT NUMBER: 134:340101

TITLE: Method development and optimization of enantiomeric

separations using macrocyclic

glycopeptide chiral stationary phases

AUTHOR(S): Beesley, Thomas E.; Lee, J. T.; Wang, Andy X.

CORPORATE SOURCE: Germany

SOURCE: Chiral Separation Techniques (2nd Edition) (2001),

25-54. Editor(s): Subramanian, Ganapathy. Wiley-VCH

Verlag GmbH: Weinheim, Germany.

CODEN: 69AXQK

DOCUMENT TYPE: Conference; General Review

LANGUAGE: English

AB A review with 39 refs.

REFERENCE COUNT: 39 THERE ARE 39 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 3 OF 4 MEDLINE on STN DUPLICATE 1

ACCESSION NUMBER: 1999305007 MEDLINE DOCUMENT NUMBER: PubMed ID: 10378662

TITLE: Methodological approaches to the analysis of IgA1

O-glycosylation in IgA nephropathy.

AUTHOR: Allen A C

CORPORATE SOURCE: Department of Nephrology, Leicester General Hospital, UK..

aa50@le.ac.uk

SOURCE: Journal of nephrology, (1999 Mar-Apr) Vol. 12, No. 2, pp.

76-84. Ref: 46

Journal code: 9012268. ISSN: 1121-8428.

PUB. COUNTRY: Italy

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

General Review; (REVIEW)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 199907

ENTRY DATE: Entered STN: 30 Jul 1999

Last Updated on STN: 30 Jul 1999 Entered Medline: 22 Jul 1999

AB IgA nephropathy (IgAN) is a common form of glomerulonephritis in which

IgAl molecules deposit in the renal mesangium, leading to progressive glomerular inflammatory injury in a significant proportion of patients. The mechanisms underlying the pathogenesis of IgAN remain poorly understood, but altered O-glycosylation, a physicochemical abnormality of IgAl observed in these patients, may be a contributory factor. Although many studies have reported aberrant IgAl O-glycosylation in IgAN, the precise structural nature of the defect remains to be fully characterised, and analysis of IgAl O-glycans has proved technically challenging. Three main strategies have been employed: lectin binding to the O-glycans in situ on the whole IgAl molecule; mass spectroscopy of isolated O-glycosylated glycopeptides; and size/charge separation of free O-glycans released from IgAl. In this review, the basic principles, strengths and weaknesses of each of these methodological approaches are considered, together with a summary of the data obtained from their use. One of the common criticisms of many studies of IqA1 O-glycosylation is the method of IgAl purification employed, and therefore, this issue is also critically discussed.

L7 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1983:213753 CAPLUS

DOCUMENT NUMBER: 98:213753

TITLE: Separation of hinge glycopeptides

of human IgD by HPLC

AUTHOR(S): Takahashi, Nobuhiro; Tetaert, Daniel; Putnam, Frank W. CORPORATE SOURCE: Dep. Biol., Indiana Univ., Bloomington, IN, 47405, USA

SOURCE: Methods Protein Sequence Anal., [Proc. Int. Conf.], 4th (1982), Meeting Date 1981, 463-70. Editor(s):

Elzinga, Marshall. Humana: Clifton, N. J.

CODEN: 49KBAY

DOCUMENT TYPE: Conference; General Review

LANGUAGE: English

AB A review with 4 refs. of the separation and amino acid sequence anal. of the human IgD hinge region using high pressure liquid chromatog. (HPLC).

=> dup rem 18

PROCESSING COMPLETED FOR L8

L9 10 DUP REM L8 (3 DUPLICATES REMOVED)

=> d ibib abs total

L9 ANSWER 1 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 2000:353053 BIOSIS DOCUMENT NUMBER: PREV200000353053

TITLE: Analysis of glycoconjugates: I. Chromatographic methods.

AUTHOR(S): Lee, Y. C. [Reprint author]

CORPORATE SOURCE: Department of Biology, Johns Hopkins University, Baltimore,

MD, 21210, USA

SOURCE: Analytical Biochemistry, (July 15, 2000) Vol. 283, No. 1,

pp. 1-2. print.

CODEN: ANBCA2. ISSN: 0003-2697.

DOCUMENT TYPE: Article

General Review; (Literature Review)

LANGUAGE: English

ENTRY DATE: Entered STN: 16 Aug 2000

Last Updated on STN: 8 Jan 2002

L9 ANSWER 2 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 1998:356832 BIOSIS DOCUMENT NUMBER: PREV199800356832

TITLE: The cytoplasmic F-box binding protein SKP1 contains a novel

pentasaccharide linked to hydroxyproline in Dictyostelium.

AUTHOR(S): Teng-Umnuay, Patana; Morris, Howard R.; Dell, Anne; Panico,

Maria; Paxton, Thanai; West, Christopher M. [Reprint

authorl

CORPORATE SOURCE: Univ. Florida Coll. Med., 1600 SW Archer Rd., Gainesville,

FL 32610-0235, USA

SOURCE: Journal of Biological Chemistry, (July 17, 1998) Vol. 273,

No. 29, pp. 18242-18249. print.

CODEN: JBCHA3. ISSN: 0021-9258.

DOCUMENT TYPE: Article LANGUAGE: English

ENTRY DATE: Entered STN: 27 Aug 1998

Last Updated on STN: 27 Aug 1998

SKP1 is involved in the ubiquitination of certain cell cycle and nutritional regulatory proteins for rapid turnover. SKP1 from Dictyostelium has been known to be modified by an oligosaccharide containing Fuc and Gal, which is unusual for a cytoplasmic or nuclear protein. To establish how it is glycosylated, SKP1 labeled with (3H) Fuc was purified to homogeneity and digested with endo-Lys-C. A single radioactive peptide was found after two-dimensional high performance liquid chromatography. Analysis in a quadrupole time-of-flight mass spectrometer revealed a predominant ion with a novel mass. Tandem mass spectrometry analysis yielded a set of daughter ions which identified the peptide and showed that it was modified at Pro-143. A second series of daughter ions showed that Pro-143 was hydroxylated and derivatized with a potentially linear pentasaccharide, HexfwdarwHexfwdarwFucfwdarwHexfwdarwHe xNAcfwdarw(HyPro). The attachment site was confirmed by Edman degradation. Gas chromatography-mass spectrometry analysis of trimethylsilyl-derivatives of overexpressed SKP1 after methanolysis showed the HexNAc to be GlcNAc. Exoglycosidase digestions of the glycopeptide from normal SKP1 and from a fucosylation mutant, followed by matrix-assisted laser desorption time-of-flight mass spectrometry analysis, showed that the sugar chain consisted of D-Galpalphalfwdarw6-D-GalpalphalfwdarwL-Fucpalphalfwdarw2-D-Galpbetalfwdarw3GlcNAc. Matrix-assisted laser-desorption time-of-flight mass spectrometry analysis of all SKP1 peptides resolved by reversed phase-high performance liquid chromatography showed that SKP1 was only partially hydroxylated at Pro-143 and that all hydroxylated SKP1 was completely glycosylated. Thus SKP1 is variably modified by an unusual linear pentasaccharide, suggesting the localization of a novel glycosylation pathway in the cytoplasm.

ANSWER 3 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 1998:413240 BIOSIS DOCUMENT NUMBER: PREV199800413240

Affinity chromatography of oligosaccharides and TITLE:

glycopeptides.

AUTHOR(S): Cummings, Richard D. [Reprint author]

CORPORATE SOURCE: Univ. Okla. Health Sci. Center, Dep. Biochem. Molecular

Biol., BSEB 329, 941 S. L. Young Blvd., Oklahoma City, OK

73190, USA

SOURCE: Matejtschuk, P. [Editor]. Practical Approach Series, (1997)

pp. 123-139. Practical Approach Series; Affinity

separations. print.

Publisher: Oxford University Press, Walton Street, Oxford OX2 6DP, England; Oxford University Press, Inc., 198 Madison Avenue, New York, New York 10016, USA. Series:

Practical Approach Series.

ISSN: 0957-025X. ISBN: 0-19-963551-X.

DOCUMENT TYPE: Book

Book; (Book Chapter)

LANGUAGE: English

Entered STN: 2 Oct 1998 ENTRY DATE:

Last Updated on STN: 2 Oct 1998

L9 ANSWER 4 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN ACCESSION NUMBER: 1996:308095 BIOSIS

DOCUMENT NUMBER: PREV199699030451

TITLE: Oligosaccharides of recombinant human alpha-L-iduronidase

secreted by Chinese hamster ovary cells.

AUTHOR(S): Zhao, K. W. [Reprint author]; Stevens, R. L.; Faull, K. F.;

Kakkis, E. D.; Neufeld, E. F.

UCLA Sch. Med., Los Angeles, CA 90095, USA CORPORATE SOURCE:

FASEB Journal, (1996) Vol. 10, No. 6, pp. Al108. SOURCE:

Meeting Info.: Joint Meeting of the American Society for Biochemistry and Molecular Biology, the American Society for Investigative Pathology and the American Association of Immunologists. New Orleans, Louisiana, USA. June 2-6, 1996.

CODEN: FAJOEC. ISSN: 0892-6638.

DOCUMENT TYPE: Conference; (Meeting)

Conference; Abstract; (Meeting Abstract)

English LANGUAGE:

ENTRY DATE: Entered STN: 2 Jul 1996

Last Updated on STN: 2 Jul 1996

ANSWER 5 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 1997:60487 BIOSIS PREV199799359690 DOCUMENT NUMBER:

Complete 1H and 13C resonance assignments of a 21-amino TITLE:

acid glycopeptide prepared from human serum transferrin.

AUTHOR(S): Lu, Jianyun; Van Halbeek, Herman [Reprint author]

CORPORATE SOURCE: Rega Inst., Dep. Med. Chem., Katholieke Univ. Leuven,

Minderbroedersstraat 10, B-3000 Leuven, Belgium

SOURCE: Carbohydrate Research, (1996) Vol. 296, No. 0, pp. 1-21.

CODEN: CRBRAT. ISSN: 0008-6215.

DOCUMENT TYPE: Article LANGUAGE: English

ENTRY DATE: Entered STN: 11 Feb 1997

Last Updated on STN: 11 Feb 1997

A 21-amino acid glycopeptide (Gp21) was isolated and purified in multi-milligram yields from commercially available human serum transferrin (HSTF) by a combination of tryptic digestion, Con A affinity chromatography, and reverse phase HPLC. The peptide chain of Gp21 contains a single N-glycosylation site to which a diantennary oligosaccharide is attached. The amino acid sequence and the glycan primary structure of Gp21 have been verified by peptide sequencing, electrospray mass spectrometry, and one-dimensional 1H NMR spectroscopy. Different glycoforms were found for the glycan of Gp21 derived from two different batches of commercial HSTF. These glycoforins differ from one another in the number of NeuAc residues (ranging from 0 to 2) and/or the number of Gal residues (ranging from 1 to 2). As for the monogalacto species, in the two-dimensional nuclear Overhauser effect (NOE) spectrum of Gp21, interglycosidic NOEs were observed between Man4 in the alpha(1 fwdarw 3) branch and the terminal GlcNAc-beta(1 fwdarw 2) residue. No interglycosidic NOE was observed between Man4 in the alpha(1 fwdarw 6) branch and the terminal GlcNAc residue. These observations indicate that the terminal GlcNAc residue in the minor glycoforms of Gp21 is exclusively located in the alpha(1 fwdarw 3) branch of the Gp21 glycan. The occurrence of such a carbohydrate structure in HSTF has not been reported before. The 1H and 13C NMR spectra of Gp21 have been completely assigned by two-dimensional homonuclear and heteronuclear spectroscopy. The close similarity of the 1H and 13C chemical shift values for the Gp21 glycan with the respective values for the peptide-free diantennary oligosaccharide (Wieruszeski et al., Glycoconjugate J., 6 (1989) 183-194) indicates that the 1H and 13C C chemical shifts of the diantennary oligosaccharide are not perturbed by the presence of the Gp21 peptide fragment. The complete 1H and 13C resonance assignments and the full characterization of the primary structure of Gp21 will permit us to study the conformation and dynamics of the N-linked diantennary oligosaccharides while covalently attached to a polypeptide fragment.

ACCESSION NUMBER: 96149843 MEDLINE DOCUMENT NUMBER: PubMed ID: 8594983

Concanavalin A- and wheat germ agglutinin-conjugated TITLE:

lectins as a tool for the identification of multiple N-glycosylation sites in heterologous protein expressed in

yeast.

AUTHOR: Garcia R; Rodriguez R; Montesino R; Besada V; Gonzalez J;

Cremata J A

CORPORATE SOURCE: GlycoLab, Bio Industry Division, Havana, Cuba.

SOURCE: Analytical biochemistry, (1995 Nov 1) Vol. 231, No. 2, pp.

342-8.

Journal code: 0370535. ISSN: 0003-2697.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 199604

ENTRY DATE: Entered STN: 22 Apr 1996

Last Updated on STN: 22 Apr 1996

Entered Medline: 9 Apr 1996

AB We report here a methodology that allows the identification of glycosylation sites by a combination of protein enzymatic digestion, glycopeptide separation on a reverse-phase HPLC column, and further recognition in a dot-blot system using concanavalin A-horseradish peroxidase. Wheat germ agglutinin-horseradish peroxidase is used as the recognition system for peptides generated after proteolytic digestion of endoglycosidase H deglycosylated protein. Glycosylation sites were confirmed by automatic Edman degradation and fast atom bombardment mass spectrometry. This methodology was applied to a model glycoprotein, alpha-amylase from Bacillus licheniformis, which is unglycosylated in its natural host and appears highly glycosylated when expressed in the methylotrophic yeast Pichia pastoris.

L9 ANSWER 7 OF 10 MEDLINE on STN DUPLICATE 2

ACCESSION NUMBER: 94297681 MEDLINE DOCUMENT NUMBER: PubMed ID: 7517757

Improved fractionation of sialylated glycopeptides by TITLE:

pellicular anion-exchange chromatography.

AUTHOR: Rohrer J S

CORPORATE SOURCE: Dionex Corporation, Sunnyvale, CA 94086.

SOURCE: Journal of chromatography. A, (1994 Apr 29) Vol. 667, No.

1-2, pp. 75-83.

Journal code: 9318488. ISSN: 0021-9673.

PUB. COUNTRY: Netherlands

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 199408

ENTRY DATE: Entered STN: 18 Aug 1994

> Last Updated on STN: 29 Jan 1996 Entered Medline: 10 Aug 1994

AB The glycoprotein bovine fetuin was treated with trypsin and the Asn-81 tryptic glycopeptide was purified (90% pure by Edman sequencing) by reversed-phase chromatography (RP-HPLC). The Asn-81 glycopeptide, which eluted as a single peak by RP-HPLC, was separable into five peaks on the NucleoPac PA100 column, a pellicular anion-exchange column. Each of the five Asn-81 glycopeptide peaks was shown to contain N-linked oligosaccharides by treatment of each peak with peptide N4-(N-acetyl-beta-D-glucosaminyl) asparagine amidase F (PNGase F) and subsequent oligosaccharide analysis by high-pH anion-exchange chromatography with pulsed amperometric detection. High-pH anion-exchange chromatography-pulsed amperometric detection oligosaccharide analysis revealed that each peak contained a different population of sialylated N-linked oligosaccharides. Hence each peak contained a different group of glycopeptide glycoforms. It was observed that the longer the retention

time of the Asn-81 glycopeptide peak on the anion-exchange column, the greater the oligosaccharide sialylation. Two glycopeptide peaks which differed in their distribution of disialylated oligosaccharides demonstrated that the glycopeptide separation was a result of something more than gross differences in sialic acid content. The two other N-linked tryptic glycopeptides of fetuin were also separated into multiple peaks on the NucleoPac PA100 column and these separations were shown to be due to differences in oligosaccharide sialylation. separations of the three fetuin N-linked glycopeptides demonstrate that pellicular anion-exchange chromatography offers improved separation speed and resolution for the separation of sialylated glycopeptides.

ANSWER 8 OF 10 MEDLINE on STN ACCESSION NUMBER: 83235063 MEDLINE DOCUMENT NUMBER: PubMed ID: 6190682

TITLE: The variant surface glycoproteins of Trypanosoma

equiperdum. Identification of a phosphorylated glycopeptide

as the cross-reacting antigenic determinant.

AUTHOR: Baltz T; Duvillier G; Giroud C; Richet C; Baltz D; Degand P SOURCE:

FEBS letters, (1983 Jul 11) Vol. 158, No. 1, pp. 174-8.

Journal code: 0155157. ISSN: 0014-5793.

PUB. COUNTRY: Netherlands

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 198308

ENTRY DATE: Entered STN: 19 Mar 1990

> Last Updated on STN: 19 Mar 1990 Entered Medline: 26 Aug 1983

AΒ The cross-reacting antigenic determinant in the variant surface glycoproteins (VSGs) of Trypanosoma equiperdum was studied by testing the ability of VSG glycopeptides to bind heterologous anti-VSG sera. VSG glycopeptide purification revealed the presence of 3 oligosaccharide sidechains on the mature VSG. These consist of two sidechains containing only mannose and glucosamine and a third containing galactose and mannose (in a 5:1 ratio) as well as phosphorous and ethanolamine. This phosphorylated fragment completely blocked the binding of VSG to heterologous anti-VSG and therefore contained the cross-reacting determinants.

ANSWER 9 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN T. 9

ACCESSION NUMBER: 1980:193704 CAPLUS

DOCUMENT NUMBER: 92:193704

TITLE: Lectins in glycopeptide separation

AUTHOR(S): Jarnefelt, Johan; Krusius, Tom; Finne, Jukka CORPORATE SOURCE: Dep. Med. Chem., Univ. Helsinki, Finland

SOURCE: Protides of the Biological Fluids (1980), Volume Date

1979, 27th, 603-6

CODEN: PBFPA6; ISSN: 0079-7065

DOCUMENT TYPE: Journal LANGUAGE: English

A method for the separation of different types of glycopeptides by chromatog. on Sepharose-linked lectins is described. Affinity chromatog. of glycopeptides was carried out on Concanavalin A (ConA)-Sepharose columns (5-10-mL bed volume) at $0-4^{\circ}$, with 0.1M NaCl in NaOAc buffer, pH 5.2,followed by 20 and 200 mM α -methylglucoside in NaOAc buffer, pH 5.2. Affinity chromatog. was also done on wheat germ agglutinin (WGA)-Sepharose, with initial buffer of 5 mM NaOAc (pH 5.2) and elution by 20 mM HCl. The structure of the eluted materials was determined by total sugar anal. and by anal. of the partially methylated alditol acetates. Fractionation of glycopeptides on Con A-Sepharose separated 4 different classes of glycopeptides on the basis of their oligosaccharide structure: fraction A glycopeptides with 3-4 branches attached to the peripheral mannose and O-glycosidically linked glycopeptides, fraction B with 2 branches, and fraction C of the oligomannoside type. Fractionation

on WGA-Sepharose produced fraction that differed in their content of fucose and 4-O-linked and 3,4-di-O-linked N-acetylglucosamine.

L9 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1971:416003 CAPLUS

DOCUMENT NUMBER: 75:16003

TITLE: Technique for extraction and purification of a

peptidoglycan from a gram-negative bacterium

AUTHOR(S): Fleck, Jacqueline; Mock, Michele

CORPORATE SOURCE: Inst. Bacteriol., Fac. Med., Strasbourg, Fr.

SOURCE: Comptes Rendus des Seances de l'Academie des Sciences,

Serie D: Sciences Naturelles (1971), 272(11), 1560-2

CODEN: CHDDAT; ISSN: 0567-655X

DOCUMENT TYPE: Journal LANGUAGE: French

AB Various methods for the extraction and purification of a peptidoglycan from Proteus vulgaris were described. The most satisfactory and rapid method was the one in which the bacteria were not disintegrated or washed, but immediately treated with boiling 4% Na dodecyl sulfate and then treated with trypsin or protease from Bacillus subtilis, especially the latter. The peptidoglycan contained 1.75 mole of alanine, 1 mole of glutamic acid, and 2.2 moles of hexosamine per 1 mole of diaminopimelic acid. Other amino acids were absent or present in very small amts.

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=> glycopeptide (3a) chromatography L10 313 GLYCOPEPTIDE (3A) CHROMATOGRAPHY

=> dup rem 111
PROCESSING COMPLETED FOR L11
L12 3 DUP REM L11 (4 DUPLICATES REMOVED)

=> d ibib abs total

L12 ANSWER 1 OF 3 MEDLINE on STN

ACCESSION NUMBER: 87051466 MEDLINE DOCUMENT NUMBER: PubMed ID: 3779692

TITLE: Glycoproteins of human teratocarcinoma cells (PA1) carry

both anomers of O-glycosyl-linked D-galactopyranosyl-(1----

3)-2-acetamido- 2-deoxy-alpha-D-galactopyranosyl group.

AUTHOR: Leppanen A; Korvuo A; Puro K; Renkonen O

SOURCE: Carbohydrate research, (1986 Sep 15) Vol. 153, No. 1, pp.

87-95.

Journal code: 0043535. ISSN: 0008-6215.

PUB. COUNTRY:

DOCUMENT TYPE:

Netherlands
Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 198701

ENTRY DATE: Entered STN: 2 Mar 1990

Last Updated on STN: 2 Mar 1990 Entered Medline: 2 Jan 1987

AB Two disaccharide alcohols, alpha-D-Galp(1----3)-GalNAcol and beta-D-Galp-(1----3)-GalNAcol, together with a GalNAcol-containing tetra-or penta-saccharide alcohol, were released from human embryonal carcinoma cells of line PA1 by reductive beta-elimination. The disaccharides were identified by exoglycosidase digestions and by periodate oxidation. The results were confirmed by affinity chromatography of the disaccharide alcohols on immobilized Bandeirea simplicifolia lectin and by chromatography of the parent glycopeptides on immobilized peanut lectin.

L12 ANSWER 2 OF 3 MEDLINE on STN DUPLICATE 2

ACCESSION NUMBER: DOCUMENT NUMBER:

86077685 MEDLINE PubMed ID: 3935160

TITLE:

Evidence for unique homologous peptide sequences around the

glycosylated seryl and threonyl residues in

polysialoglycoproteins isolated from the unfertilized eggs

of the Pacific salmon Oncorhynchus keta.

AUTHOR:

Shimamura M; Inoue Y; Inoue S

SOURCE: Biochemistry, (1985 Sep 24) Vol. 24, No. 20, pp. 5470-80.

Journal code: 0370623. ISSN: 0006-2960.

PUB. COUNTRY: United States

DOCUMENT TYPE:

Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT:

Priority Journals

ENTRY MONTH:

198602

ENTRY DATE:

Entered STN: 21 Mar 1990

Last Updated on STN: 21 Mar 1990

Entered Medline: 7 Feb 1986

AB Structures of glycopeptides obtained by exhaustive Pronase digestion of high molecular weight (1.7 X 10(5)) salmon egg polysialoglycoprotein has

high molecular weight (1.7 X 10(5)) salmon egg polysialoglycoprotein have been elucidated. Six principal glycopeptides isolated by gel chromatography and DEAE-Sephadex A-25 chromatography in the absence or presence of borate ion were analyzed for their carbohydrate and amino acid composition, as well as amino acid sequence, and found to be of two distinct types: glycotripeptides, Thr*-Ser*-Glu, and glycotetrapeptides, Thr*-Gly-Pro-Ser, where an asterisk indicates the amino acid residues to which either the Gal beta 1----3GalNAc or Fuc alpha 1----3GalNAc beta 1----3Gal beta 1----4Gal beta 1----3GalNAc chain is attached. Their final yield corresponds to 64% of the original desialylated glycoprotein. In view of the simple amino acid composition of salmon egg polysialoglycoprotein (molar ratio Asp2Thr2Ser3Glu1Pro1Gly1Ala3) and the result of alkaline betaelimination indicating three carbohydrate units linked to two of two threonine and one of three serine residues, a unique primary structure comprising repetitive sequences of the above two types of glycopeptides, which are interspersed by short nonglycosylated peptides consisting of alanine and aspartic acid, has been proposed for the core protein. The molecular secondary ion mass spectra of underivatized glycopeptides were

used to obtain their structural information. The anomeric configuration of the proximal sugar-peptide linkages was proven to be alpha by proton nuclear magnetic resonance spectroscopy. This is the first systematic reported study of O-glycosidically linked glycopeptides by these instrumental methods.

L12 ANSWER 3 OF 3 MEDLINE on STN DUPLICATE 3

ACCESSION NUMBER: 85120881 MEDLINE DOCUMENT NUMBER: PubMed ID: 2982317

TITLE: Galactose-rich glycoproteins are on the cell surface of

herpes virus-infected cells. 1. Surface labeling and serial lectin binding studies of Asn-linked oligosaccharides of

glycoprotein gC.

AUTHOR: Kumarasamy R; Blough H A

SOURCE: Archives of biochemistry and biophysics, (1985 Feb 1) Vol.

236, No. 2, pp. 593-602.

Journal code: 0372430. ISSN: 0003-9861.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 198503

ENTRY DATE: Entered STN: 20 Mar 1990

Last Updated on STN: 20 Mar 1990 Entered Medline: 21 Mar 1985

Cell-surface glycoproteins of mock-infected and herpes simplex virus type AΒ 1 (HSV-1)-infected BHK-21 and HEp-2 cells were radiolabeled by incubation with galactose oxidase followed by reduction with NaB3H4. The incorporation of radiolabel into glycoconjugates in both BHK-21 and HEp-2 cells was increased several fold following infection with HSV, showing an increase in surface-exposed Gal residues in the infected cells. This was further confirmed by an increase in binding of cell-surface-labeled glycoproteins gC and gB from HSV-infected BHK-21 cells to Ricinus communis agglutinin I, which is specific for beta-D-Gal residues. Prior treatment of cells with Clostridium perfringens neuraminidase enhanced the surface radiolabeling by the galactose oxidase/NaB3H4 method: HEp-2 cells exhibited over sixfold enhancement in labeling, while BHK-21 cells showed only a slight increase. HSV glycoprotein gC was the predominant cell-surface glycoprotein radiolabeled by the galactose oxidase/NaB3H4 method in virus-infected BHK-21 cells. The glycoprotein gC was purified by immunoaffinity column chromatography on monoclonal anti-gC-antibody-Sepharose. The radiolabel in the glycopeptides of gC was resistant to beta elimination, showing that it was associated only with Asn-linked oligosaccharides. A serial lectin affinity chromatography of glycopeptides on columns of concanavalin A-Sepharose, lentil (Lens culinaris) lectin-Sepharose, and Ricin I-agarose allowed the assignment of minimal oligosaccharide structures bearing terminal Gal residues in gC.

=> dup rem 113

PROCESSING COMPLETED FOR L13

L14 10 DUP REM L13 (8 DUPLICATES REMOVED)

=> d ibib abs total

L14 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 1

ACCESSION NUMBER: 2005:922933 CAPLUS

DOCUMENT NUMBER: 143:386432

TITLE: Solid-phase synthesis of hydroxypiperazine derivatives

using phenethylamine linker by oxidation-Cope

elimination

AUTHOR(S): Seo, Jin-soo; Kim, Hye-won; Yoon, Cheol Min; Ha, Deok

Chan; Gong, Young-Dae

CORPORATE SOURCE: Medicinal Science Division, Korea Research Institute

of Chemical Technology, Yusung-gu, Daejeon, 305-600,

S. Koreà

SOURCE: Tetrahedron (2005), 61(39), 9305-9311

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER: Elsevier B.V.

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 143:386432

AB A general method is reported for the parallel solid-phase synthesis of hydroxypiperazine derivs. based on the oxidation-Cope elimination of polymer-bound phenethylamine linker with m-CPBA. The key intermediate of

phenethylamine N-oxide resins was separable on solid-

phase for subsequent β -elimination, from

which the desired hydroxypiperazine products could be obtained in high purities and yields without any significant contamination at 90 °C for 2 h. The utility of the methodol. for solid-phase synthesis of general hydroxylamines was also investigated using the same linker. The progress of reactions could be monitored on polymer bound intermediates by ATR-FTIR spectroscopy on single bead. The desired products were obtained in good six-step overall yields upon cleavage from the resins and were

characterized by LC/MS, 1H NMR, and 13C NMR spectroscopy.

REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L14 ANSWER 2 OF 10 MEDLINE on STN DUPLICATE 2

ACCESSION NUMBER: 2005381769 MEDLINE DOCUMENT NUMBER: PubMed ID: 16039524

TITLE: Phosphoproteomic analysis with a solid-phase

capture-release-tag approach.

AUTHOR: Tseng Huang-Chun; Ovaa Huib; Wei Nancy J C; Ploegh Hidde;

Tsai Li-Huei

CORPORATE SOURCE: Howard Hughes Medical Institute, Harvard Medical School,

Boston, Massachusetts 02155, USA.. huang-

chun tseng@hms.harvard.edu

SOURCE: Chemistry & biology, (2005 Jul) Vol. 12, No. 7, pp. 769-77.

Journal code: 9500160. ISSN: 1074-5521.

PUB. COUNTRY: England: United Kingdom

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 200510

ENTRY DATE: Entered STN: 26 Jul 2005

Last Updated on STN: 28 Oct 2005 Entered Medline: 27 Oct 2005

AB A comprehensive study of global phosphorylation events in biological systems is critical. We report a chemistry-based capture-release-tag method for isolation of complex phospho-Ser/Thr-containing peptides by liquid beta-elimination combined with solid-phase Michael addition. The free thiol groups of 6-(mercapto-acetylamino)-hexanoic acid functionalized resin are used as immobilized Michael donors to capture dehydro-serine/threonine peptides. After an acid-mediated release step, phospho-peptides are labeled with a 6-(2-mercapto-acetylamine)-hexanoic amide tag at phosphorylated sites. We applied this method to analyze the phosphorylation status of microtubule-associated proteins. We find that a CDK5 substrate microtubule-associated protein 2 (MAP2) is phosphorylated on residues that are within a homologous region of Tau. The chemical method corroborates previous results and suggests that Tau and MAP2 may contain a CDK5 phosphorylation motif.

L14 ANSWER 3 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN ACCESSION NUMBER: 2005:395356 BIOSIS

DOCUMENT NUMBER: PREV200510185388

TITLE: Comparative phosphoproteomic analysis of synaptosomal

proteins of a progressive neurodegeneration mouse model. Tseng, Huang-Chun [Reprint Author]; Ovaa, Huib; Chang,

AUTHOR(S): Tseng, Huang-Chun [Reprint Author]; Nancy; Ploegh, Hidde; Tsai, Li-Huei

CORPORATE SOURCE: Howard Hughes Med Inst, Boston, MA 02115 USA

SOURCE: FASEB Journal, (MAY 14 2004) Vol. 18, No. 8, Suppl. S, pp.

C61.

Meeting Info.: Annual Meeting of the American-Society-for-Biochemistry-and-Molecular-Biology/8th Congress of the International-Union-for-Biochemistry-and-Molecular-Biology. Boston, MA, USA. June 12 -16, 2004. Amer Soc BioChem & Mol

Biol; Int Union Biochem & Mol Biol. CODEN: FAJOEC. ISSN: 0892-6638.

DOCUMENT TYPE: Conference; (Meeting)

Conference; Abstract; (Meeting Abstract)

LANGUAGE: English

ENTRY DATE: Entered STN: 5 Oct 2005

Last Updated on STN: 5 Oct 2005

Aberrent kinase activities in brain can lead to neurodegeneration. We use a novel chemistry-based capture-release-tag method for the isolation of phospho-Ser/Thr-containing peptides by beta-elimination followed by a solid-phase Michael addition. After the chemistry, phosphate at the phosphorylation sites will be replaced a specific chemical tag. Tagged serines and threonines can then be identified by tandem mass spectrometry. We analyze phosphorylation sites of synaptosomal proteins prepared from control brains and p25(Cdk5 activator)transgenic mouse brains with different degree of neurodegeneration. Results from phosphopeptide clustering analysis reveal several distinct groups of phosphorylation, suggesting changes of specific kinase/phosphase activities during neurodegeneration. Several novel

L14 ANSWER 4 OF 10 MEDLINE on STN DUPLICATE 3

ACCESSION NUMBER: 2003424036 MEDLINE DOCUMENT NUMBER: PubMed ID: 12964774

TITLE: Characterization of protein phosphorylation by mass spectrometry using immobilized metal ion affinity

hypothesis are created, based the phosphopeptide cluster data.

chromatography with on-resin .

beta-elimination and Michael addition.

AUTHOR: Thompson Andrew J; Hart Sarah R; Franz Clemens; Barnouin

Karin; Ridley Anne; Cramer Rainer

CORPORATE SOURCE: The Ludwig Institute for Cancer Research, Cruciform

Building, Gower Street, London WC1E 6BT, United Kingdom. Analytical chemistry, (2003 Jul 1) Vol. 75, No. 13, pp.

3232-43.

SOURCE:

Journal code: 0370536, ISSN: 0003-2700.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 200312

ENTRY DATE: Entered STN: 11 Sep 2003

Last Updated on STN: 18 Dec 2003 Entered Medline: 10 Dec 2003

AB A protocol combining immobilized metal ion affinity chromatography and beta-elimination with concurrent Michael addition has been developed for enhanced analysis of protein phosphorylation. Immobilized metal ion affinity chromatography was initially used to enrich for phosphorylated peptides. Beta-elimination, with or without concurrent Michael addition, was then subsequently used to simultaneously elute and derivatize phosphopeptides bound to the chromatography resin. Derivatization of the phosphate facilitated the precise determination of phosphorylation sites by MALDI-PSD/LIFT tandem mass spectrometry, avoiding complications due to ion suppression and phosphate lability in mass spectrometric analysis of

phosphopeptides. Complementary use of immobilized metal ion affinity chromatography and beta-elimination with concurrent Michael addition in this manner circumvented several inherent disadvantages of the individual methods. In particular, (i) the protocol discriminated O-linked glycosylated peptides from phosphopeptides prior to beta-elimination/Michael addition and (ii) the elution of peptides from the chromatography resin as derivatized phosphopeptides distinguished them from unphosphorylated species that were also retained. The chemical derivatization of phosphopeptides greatly increased the information obtained during peptide sequencing by mass spectrometry. The combined protocol enabled the detection and sequencing of phosphopeptides from protein digests at low femtomole concentrations of initial sample and was employed to identify novel phosphorylation sites on the cell adhesion protein pl20 catenin and the glycoprotein fetuin.

L14 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2003:573681 CAPLUS

DOCUMENT NUMBER: 139:381153

TITLE: Selenium-linking strategy for traceless solid-phase

synthesis of acrylamides

AUTHOR(S): Sheng, Shou-Ri; Wang, Xing-Cong; Liu, Xiao-Ling; Song,

Cai-Sheng

CORPORATE SOURCE: Department of Chemistry, Jiangxi Normal University,

Nanchang, Peop. Rep. China

SOURCE: Synthetic Communications (2003), 33(16), 2867-2872

CODEN: SYNCAV; ISSN: 0039-7911

PUBLISHER: Marcel Dekker, Inc.

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 139:381153

AB A novel polystyrene-supported β -selenopropionic acid was prepared and

applied to simple and efficient synthesis of acrylamides.

Polystyrene-supported selenium bromide was metalated by NaBH4 and reacted with 3-bromopropionic acid to give supported 3-selenylpropionic acid, which was converted to acyl chloride and reacted with R1R2NH (R2 = H, R1 = Ph, 4-MeC6H4, 4-FC6H4, 4-ClC6H4, 4-BrC6H4, PhCH2, Bu; R1 = R2 = iPr;

R1R2NH = piperidine) to give corresponding amides. The H2O2 oxidation of the

amides with subsequent β -elimination gave substituted acrylamides.

REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L14 ANSWER 6 OF 10 MEDLINE on STN DUPLICATE 4

ACCESSION NUMBER: 2002444688 MEDLINE DOCUMENT NUMBER: PubMed ID: 12201785

TITLE: Versatile "traceless" sulfone linker for SPOS: preparation

of isoxazolinopyrrole 2-carboxylates.

AUTHOR: Hwang Sung Hee; Kurth Mark J

CORPORATE SOURCE: Department of Chemistry, University of California, One

Shields Avenue, Davis, California 95616-5295, USA.

SOURCE: The Journal of organic chemistry, (2002 Sep 6) Vol. 67, No.

18, pp. 6564-7.

Journal code: 2985193R. ISSN: 0022-3263.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: NONMEDLINE; PUBMED-NOT-MEDLINE

ENTRY MONTH: 200304

ENTRY DATE: Entered STN: 31 Aug 2002

Last Updated on STN: 6 Apr 2003 Entered Medline: 4 Apr 2003

AB A five-step solid-phase synthesis of isoxazolinopyrrole-2-carboxylates (6) that employs a traceless sulfone linker strategy is reported. Resin-bound diene 4, obtained by acetylation and concomitant beta-elimination of acetate from resin-bound allylic alcohol 3, underwent regioselective 1,3-dipolar cycloadditons with nitrile oxides.

Formation of the pyrrole products in a resin-releasing strategy was performed by pyrrole annulation with alkyl isocyanoacetates, which react with the vinyl sulfone moiety to generate the target isoxazolinopyrrole-2-carboxylates (6). Use of this chemistry afforded eight isoxazolinopyrrole-2-carboxylates in 6-24% overall yields from polystyrene/divinylbenzene sulfinate 1.

L14 ANSWER 7 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 2003:54648 BIOSIS DOCUMENT NUMBER: PREV200300054648

TITLE: Improved enrichment of phosphopeptides by IMAC with on-

resin beta-elimination and

Michael addition.

AUTHOR(S): Thompson, Andrew; Hart, Sarah; Barnouin, Karin; Cramer,

Rainer

SOURCE: Molecular & Cellular Proteomics, (September 2002) Vol. 1,

No. 9, pp. 698. print.

Meeting Info.: First World Congress of the Human Proteome Organisation. Versailles, Paris, France. November 21-24,

2002. Human Proteome Organisation. ISSN: 1535-9476 (ISSN print).

DOCUMENT TYPE: Conference; (Meeting)

Conference; Abstract; (Meeting Abstract)

LANGUAGE: English

ENTRY DATE: Entered STN: 22 Jan.2003

Last Updated on STN: 22 Jan 2003

L14 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2001:640874 CAPLUS

TITLE: Traceless sulfone linker in solid phase synthesis: Preparation of 3,5-disubstituted cyclopent-2-enone

AUTHOR(S): Kurth, Mark J.; Cheng, Wei-Chieh

CORPORATE SOURCE: Department of Chemistry, University of California,

Davis, Davis, CA, 95616, USA

SOURCE: Abstracts of Papers, 222nd ACS National Meeting,

Chicago, IL, United States, August 26-30, 2001 (2001), ORGN-511. American Chemical Society: Washington, D.

C.

CODEN: 69BUZP

DOCUMENT TYPE: Conference; Meeting Abstract

LANGUAGE: English

AB The preparation of functionalized 3,5-disubstituted cyclopent-2-enone via a traceless solid-phase sulfone linker strategy is described. Polystyrene/divinylbenzene sulfinate 1 undergoes S-alkylation followed by

 α, α -dialkyltion with 1,4-dichloro-2-butene to form polymer-bound 3-phenylsulfonylcyclopentene species. Subsequent epoxidn. was accomplished by treating mCPBA. Oxirane moiety of resin 11 was opened with various nucleophiles such as Gringerd regent, higher order Cuprate

reagent, azide ion, and amines. To complete a traceless linker cleavage strategy, swern oxidation was employed to generate polymer-bound

3-phenylsulfonylcyclopentanes and simultaneous release 3,5-disubstituted

cyclopent-2-enones from the resin via β -

elimination of the carbonyl moiety. Twelve 3,5-disubstituted

cyclopent-2-enones were prepared in five steps and 20-42% overall yield from polystyrene/divinylbenzene sulfinate 1.

L14 ANSWER 9 OF 10 MEDLINE on STN DUPLICATE 5

ACCESSION NUMBER: 96016037 MEDLINE DOCUMENT NUMBER: PubMed ID: 7546982

TITLE: Synthesis of an N-methyldehydroalanine-containing fragment

of microcystin by combination of solid

phase peptide synthesis and beta-

elimination in solution.

AUTHOR: Zetterstrom M; Trogen L; Hammarstrom L G; Juhlin L; Nilsson

B; Damberg C; Bartfai T; Langel U

CORPORATE SOURCE: Department of Neurochemistry and Neurotoxicology, Arrhenius

Laboratories, Stockholm University, Sweden.

SOURCE: Acta chemica Scandinavica (Copenhagen, Denmark: 1989),

(1995 Sep) Vol. 49, No. 9, pp. 696-700. Journal code: 9012772. ISSN: 0904-213X.

PUB. COUNTRY: Denmark

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 199510

ENTRY DATE: Entered STN: 27 Dec 1995

Last Updated on STN: 27 Dec 1995 Entered Medline: 27 Oct 1995

AΒ A new method for the synthesis of dehydroalanine (delta Ala)-containing peptides has been developed by combining solid phase peptide synthesis (tert-butyloxycarbonyl/HF-chemistry) with solution synthesis. A sequence from cyanobacterial hepatotoxin microcystin, Ac-D-gamma-Glu-[N-Me-delta Ala]-D-Ala-Leu amide was chosen as a model peptide. The precursor for the synthesis of the dehydroalanine-containing peptide, Ac-D-gamma-Glu-[N,SdiMeCys]-D-Ala-Leu, was synthesized on a solid phase followed by sulfonium salt formation on the resin. The resulting S,S-dimethylated peptide was cleaved from the resin with liquid HF. The HPLC-purified S,S-dimethylated cysteine-containing precursor peptide was subjected to beta-elimination in solution catalysed by DBU (1,8-diazabicyclo[5.4.0]undec-7-ene) in methanol. The final product, Ac-D-gamma-Glu-[N-Me-delta Ala]-D-Ala-Leu amide, was purified by HPLC, and analysed by mass spectrometry and 1H NMR spectroscopy. The stability of the model peptide under acidic, neutral and basic conditions has been studied.

L14 ANSWER 10 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on

ACCESSION NUMBER: 1991:450915 BIOSIS

DOCUMENT NUMBER: PREV199192095695; BA92:95695

TITLE: CHEMOSELECTIVE ONE-STEP PURIFICATION METHOD FOR PEPTIDES

SYNTHESIZED BY THE SOLID-PHASE TECHNIQUE.

AUTHOR(S): FUNAKOSHI S [Reprint author]; FUKUDA H; FUJII N

CORPORATE SOURCE: FAC PHARMACEUTICAL SCI, KYOTO UNIV, SAKYO-KU, KYOTO 606,

JAPAN

SOURCE: Proceedings of the National Academy of Sciences of the

United States of America, (1991) Vol. 88, No. 16, pp.

6981-6985.

CODEN: PNASA6. ISSN: 0027-8424.

DOCUMENT TYPE: Article FILE SEGMENT: BA LANGUAGE: ENGLISH

ENTRY DATE: Entered STN: 11 Oct 1991

Last Updated on STN: 11 Oct 1991

The specific reaction between SH and iodoacetamide groups has been AΒ explored as the basis of an affinity-type purification procedure for peptides synthesized by the solid-phase technique. For this affinity-type purification procedure, we synthesized an SH precursor reagent bearing an acid-labile S-protecting group, pMB-SCH2CONHCH2CH2SO2CH2CH2OCO2pNP (compound I), in which pMB is p-methoxybenzyl and pNP is p-nitrophenyl. Using this reagent, the procedure involves the following sequence of four reactions: (i) attachment of the SH function of compound I to the α -amino group of a peptide-resin through a base-labile sulfonylethoxycarbonyl linkage in the final step of solid-phase peptide synthesis, (ii) acid treatment to remove the S-pMB and side-chain-protecting groups employed and cleave the modified peptide from the resin, (iii) immobilization of the derived SH-peptide on an iodoacetamide-resin column, and (iv) base (5% NH4OH) treatment to release the desired peptide from the resin in nearly pure form. To facilitate this purification procedure, unreacted amino groups were acetylated in each step during solid-phase synthesis. The usefulness of this method was demonstrated by the purification of several peptides (18 to \approx 44

amino acids in length) synthesized by the 9-fluorenylmethoxycarbonyl (Fmoc)-based solid-phase technique. The principle of this affinity-type purification procedure may also be applied to the tert-butoxycarbonyl (Boc)-based solid-phase technique.

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Ion Exchange Chromatography (IEC)

Principals | Considerations | Buffer Systems | Vendors |

Principals

Ion Exchange Chromatography relies on charge-charge interactions between the proteins in your sample and the charges immobilized on the resin of your choice. Ion exchange chromatography can be subdivided into cation exchange chromatography, in which positively charged ions bind to a negatively charged resin; and anion exchange chromatography, in which the binding ions are negative, and the immobilized functional group is positive. Once the solutes are bound, the column is washed to equilibrate it in your starting buffer, which should be of low ionic strength, then the bound molecules are eluted off using a gradient of a second buffer which steadily increases the ionic strength of the eluent solution. Alternatively, the pH of the eluent buffer can be modified as to give your protein or the matrix a charge at which they will not interact and your molecule of interest elutes from the resin. If you know the pH you want to run at and need to decide what type of ion exchange to use paste your protein sequence into the titration curve generator. If it is negatively charged at the pH you wish, use an anion exchanger; if it is positive, use a cation exchanger. Of course this means that your protein will be binding under the conditions you choose. In many cases it may be more advantageous to actually select conditions at which your protein will flow through while the contaminants will bind. This mode of binding is often referred to as "flow through mode". This is a particularly good mode to use in the case of anion exchange. Here one could use this type of mode to bind up endotoxins or other highly negatively charged substances well at the same time relatively simply flowing your protein through the matrix.

Considerations

Anion Exchange Chromatography (AEC)

The surface charge of the solutes (proteins, nucleic acids, endotoxin) which bind will be net negative, thus to get binding of a specific protein one should be above the pl of that protein. Commonly used anion exchange resins are Q-resin, a Quaternary amine; and DEAE resin, DiEthylAminoEthane (see figure below). AEC is often used as a primary chromatography

$$\begin{array}{c} \text{CH}_3 \\ \text{Rem} \\ -\text{N} \\ \text{CH}_3 \\ \text{CH}_3 \\ \text{CH}_3 \\ \text{Q-anion exchanger} \end{array} \quad \begin{array}{c} \text{CH}_{\bar{2}} \text{ CH}_3 \\ \text{Rem} \\ -\text{CH}_{\bar{2}} \text{ CH}_2 \\ \text{CH}_2 \\ \text{CH}_3 \\ \text{CH}_2 \text{CH}_3 \\ \text{CH}_3 \\ \text{CH}_2 \text{CH}_3 \\ \text{$$

step due to its high capacity, (Matrices can bind from 10 to 100 mg of protein per ml) and ability to bind up and separate fragmented nucleic acids and lipopolysaccharides from the initial slurry. Typically, AEC is performed using buffers at pH's between 7 and 10 and running a gradient from a solution containing just this buffer to a solution containing this buffer with 1M NaCl. The salt in the solution competes for binding to the immobilized matrix and releases the protein from its bound state at a given concentration. Proteins separate because the amount of salt needed to compete varies with the external charge of the protein. Uses of AEC include initial clean up of a crude slurry, separation of proteins from each other, concentrating a protein, and the removal of negatively charged endotoxin from protein preparations.

Cation Exchange Chromatography (CEC)

The surface charge of the solutes (proteins, nucleic acids, endotoxin) which bind will be net positive, thus to get binding of a specific protein one should be below the pl of that protein. Commonly used cation exchange resins are S-resin, sulfate derivatives; and CM resins, carboxylate derived ions (see figure below).



S-cation exchanger

CM-cation exchanger

CEC is less commonly used compared to AEC, largely due to the fact that often proteins do not stick to this resin at physiological pHs and one is reluctant to titrate a protein through its isoelectric point to get it to adhere to the resin. Nonetheless, it is as powerful as AEC for initial separations with equivalently high capacity. Typically, CEC is performed using buffers at pH's between 4 and 7 and running a gradient from a solution containing just this buffer to a solution containing this buffer with 1M NaCl. Uses of CEC include initial clean up of a crude slurry, separation of proteins from each other, concentrating a protein, and as a common first purification step for proteins expressed under acidic conditions such as in *P. pastoris*.

Buffers and Buffer Systems

1. Buffers for anion exchange chromatography

Molecule	pKa	dpKa/degree C.	Counter ion
N-methyl piperazine	4.75	-0.015	chloride
piperazine	5.68	-0.015	chloride or formate
L-histidine	5.96		chloride
bis-Tris	6.46	-0.017	chloride
bis-Tris propane	6.80		chloride
triethanolamine	7.76	-0.020	chloride or acetate
Tris	8.06	-0.028	chloride
N-methyl-diethanolamine	8.52	-0.028	chloride
diethanolamine	8.88	-0.025	chloride
1,3-diaminopropane	8.64	-0.031	chloride
ethanolamine	9.50	-0.029	chloride
piperazine	9.73	-0.026	chloride
1,3-diaminopropane	10.47	-0.026	chloride
piperidine	11.12	-0.031	chloride
phosphate	12.33	-0.026	chloride

^{*}These values were taken from the Pharmacia biotech "Ion exchange chromatography, principles and methods" guidebook.

2. Buffers for cation exchange chromatography:

Molecule	рКа	dpKa/degree C.	Counter ion
Maleic acid	2.00		sodium
Malonic acid	2.88		sodium
citric acid	3.13	-0.0024	sodium
lactic acid	3.81		sodium

formic acid	3.75	0.0002	sodium or lithium
butaneandioic acid	4.21	-0.0018	sodium
acetic acid	4.76	0.0002	sodium or lithium
malonic acid	5.68		sodium or lithium
phosphate	7.20	-0.0028	sodium
HEPES	7.55	-0.0140	sodium or lithium
BICINE	8.35	-0.0180	sodium ·

^{*}These values were taken from the Pharmacia biotech "Ion exchange chromatography, principles and methods" guidebook.

Buffer system 1

Buffer A = 20 mM Tris, pH=8.0

Buffer B = 20 mM Tris, 1 M NaCl, pH=8.0

Equilibrate column in buffer A, cycle once through buffer B. Bind protein dialyzed against buffer A. Elute with a linear gradient of A to 100% B.

Buffer system 2 (Common CEC buffer system)

Buffer A = 30 mM sodium acetate, pH=4.5

Buffer B = 30 mM sodium acetate, 1 M NaCl, pH=4.5

Treat as above.

Buffer system 3 (AEC for proteins which are very insoluble or have a very high pl)

Buffer A = 30 mM Ethanolamine, 8M urea, pH=10.0

Buffer B = 30 mM Ethanolamine, 8M urea, 1 M NaCl, pH=10.0

Treat as above

Vendors

Amersham Biosciences (former Pharmacia) has a number of commonly used resins for ion exchange <u>in this list</u> or try BioRad resins <u>here.</u>

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=> glycopeptide (3a) chromatography . L10 313 GLYCOPEPTIDE (3A) CHROMATOGRAPHY

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L12 ANSWER 1 OF 3 MEDLINE on STN DUPLICATE 1

ACCESSION NUMBER: 87051466 MEDLINE DOCUMENT NUMBER: PubMed ID: 3779692

TITLE: Glycoproteins of human teratocarcinoma cells (PA1) carry

both anomers of O-glycosyl-linked D-galactopyranosyl-(1----

3)-2-acetamido- 2-deoxy-alpha-D-galactopyranosyl group.

AUTHOR: Leppanen A; Korvuo A; Puro K; Renkonen O

SOURCE: Carbohydrate research, (1986 Sep 15) Vol. 153, No. 1, pp.

87-95.

Journal code: 0043535. ISSN: 0008-6215.

PUB. COUNTRY: Netherlands

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 198701

ENTRY DATE: Entered STN: 2 Mar 1990

Last Updated on STN: 2 Mar 1990 Entered Medline: 2 Jan 1987

AB Two disaccharide alcohols, alpha-D-Galp(1---3)-GalNAcol and beta-D-Galp-(1---3)-GalNAcol, together with a GalNAcol-containing tetra-or penta-saccharide alcohol, were released from human embryonal carcinoma cells of line PA1 by reductive beta-elimination. The disaccharides were identified by exoglycosidase digestions and by periodate oxidation. The results were confirmed by affinity chromatography of the disaccharide alcohols on immobilized Bandeirea simplicifolia lectin and by chromatography of the parent glycopeptides on immobilized peanut lectin.

L12 ANSWER 2 OF 3 MEDLINE on STN DUPLICATE 2

ACCESSION NUMBER: 86077685 MEDLINE DOCUMENT NUMBER: PubMed ID: 3935160

TITLE: Evidence for unique homologous peptide sequences around the

glycosylated seryl and threonyl residues in

polysialoglycoproteins isolated from the unfertilized eggs

of the Pacific salmon Oncorhynchus keta.

AUTHOR: Shimamura M; Inoue Y; Inoue S

SOURCE: Biochemistry, (1985 Sep 24) Vol. 24, No. 20, pp. 5470-80.

Journal code: 0370623. ISSN: 0006-2960.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 198602

ENTRY DATE: Entered STN: 21 Mar 1990

Last Updated on STN: 21 Mar 1990 Entered Medline: 7 Feb 1986

AB Structures of glycopeptides obtained by exhaustive Pronase digestion of high molecular weight (1.7 X 10(5)) salmon egg polysialoglycoprotein have been elucidated. Six principal glycopeptides isolated by gel chromatography and DEAE-Sephadex A-25 chromatography in the absence or presence of borate ion were analyzed for their carbohydrate and amino acid composition, as well as amino acid sequence, and found to be of

two distinct types: glycotripeptides, Thr*-Ser*-Glu, and glycotetrapeptides, Thr*-Gly-Pro-Ser, where an asterisk indicates the amino acid residues to which either the Gal beta 1----3GalNAc or Fuc alpha 1----3GalNAc beta 1----3Gal beta 1----4Gal beta 1----3GalNAc chain is attached. Their final yield corresponds to 64% of the original desialylated glycoprotein. In view of the simple amino acid composition of salmon egg polysialoglycoprotein (molar ratio Asp2Thr2Ser3GlulProlGly1Ala3) and the result of alkaline betaelimination indicating three carbohydrate units linked to two of two threonine and one of three serine residues, a unique primary structure comprising repetitive sequences of the above two types of glycopeptides, which are interspersed by short nonglycosylated peptides consisting of alanine and aspartic acid, has been proposed for the core protein. The molecular secondary ion mass spectra of underivatized glycopeptides were used to obtain their structural information. The anomeric configuration of the proximal sugar-peptide linkages was proven to be alpha by proton nuclear magnetic resonance spectroscopy. This is the first systematic reported study of O-glycosidically linked glycopeptides by these instrumental methods.

L12 ANSWER 3 OF 3 MEDLINE on STN DUPLICATE 3

ACCESSION NUMBER: 85120881 MEDLINE DOCUMENT NUMBER: PubMed ID: 2982317

TITLE: Galactose-rich glycoproteins are on the cell surface of

herpes virus-infected cells. 1. Surface labeling and serial lectin binding studies of Asn-linked oligosaccharides of

glycoprotein gC.

AUTHOR: Kumarasamy R; Blough H A

SOURCE: Archives of biochemistry and biophysics, (1985 Feb 1) Vol.

236, No. 2, pp. 593-602.

Journal code: 0372430. ISSN: 0003-9861.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 198503

ENTRY DATE: Entered STN: 20 Mar 1990

Last Updated on STN: 20 Mar 1990 Entered Medline: 21 Mar 1985

AΒ Cell-surface glycoproteins of mock-infected and herpes simplex virus type 1 (HSV-1)-infected BHK-21 and HEp-2 cells were radiolabeled by incubation with galactose oxidase followed by reduction with NaB3H4. The incorporation of radiolabel into glycoconjugates in both BHK-21 and HEp-2 cells was increased several fold following infection with HSV, showing an increase in surface-exposed Gal residues in the infected cells. This was further confirmed by an increase in binding of cell-surface-labeled glycoproteins qC and qB from HSV-infected BHK-21 cells to Ricinus communis agglutinin I, which is specific for beta-D-Gal residues. Prior treatment of cells with Clostridium perfringens neuraminidase enhanced the surface radiolabeling by the galactose oxidase/NaB3H4 method: HEp-2 cells exhibited over sixfold enhancement in labeling, while BHK-21 cells showed only a slight increase. HSV glycoprotein gC was the predominant cell-surface glycoprotein radiolabeled by the galactose oxidase/NaB3H4 method in virus-infected BHK-21 cells. The glycoprotein gC was purified by immunoaffinity column chromatography on monoclonal anti-gC-antibody-Sepharose. The radiolabel in the glycopeptides of gC was resistant to beta elimination, showing that it was associated only with Asn-linked oligosaccharides. A serial lectin affinity chromatography of glycopeptides on columns of concanavalin A-Sepharose, lentil (Lens culinaris) lectin-Sepharose, and Ricin I-agarose allowed the assignment of minimal oligosaccharide structures bearing terminal Gal residues in qC.

=> dup rem 113

PROCESSING COMPLETED FOR L13

L14 10 DUP REM L13 (8 DUPLICATES REMOVED)

=> d ibib abs total

L14 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 1

ACCESSION NUMBER: 2005:922933 CAPLUS

DOCUMENT NUMBER: 143:386432

TITLE: Solid-phase synthesis of hydroxypiperazine derivatives

using phenethylamine linker by oxidation-Cope

elimination

AUTHOR(S): Seo, Jin-soo; Kim, Hye-won; Yoon, Cheol Min; Ha, Deok

Chan; Gong, Young-Dae

CORPORATE SOURCE: Medicinal Science Division, Korea Research Institute

of Chemical Technology, Yusung-gu, Daejeon, 305-600,

S. Korea

SOURCE: Tetrahedron (2005), 61(39), 9305-9311

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER: Elsevier B.V.

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 143:386432

AB A general method is reported for the parallel solid-phase synthesis of

hydroxypiperazine derivs. based on the oxidation-Cope elimination of polymer-bound phenethylamine linker with m-CPBA. The key intermediate of phenethylamine N-oxide resins was separable on solid-

phase for subsequent .beta.-elimination, from which the desired hydroxypiperazine products could be obtained in high purities and yields without any significant contamination at 90 °C for 2 h. The utility of the methodol. for solid-phase synthesis of general hydroxylamines was also investigated using the same linker. The progress of reactions could be monitored on polymer bound intermediates by ATR-FTIR spectroscopy on single bead. The desired products were obtained in good six-step overall yields upon cleavage from the resins and were characterized by LC/MS, 1H NMR, and 13C NMR spectroscopy.

REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L14 ANSWER 2 OF 10 MEDLINE on STN DUPLICATE 2

ACCESSION NUMBER: 2005381769 MEDLINE DOCUMENT NUMBER: PubMed ID: 16039524

TITLE: Phosphoproteomic analysis with a solid-phase

capture-release-tag approach.

AUTHOR: Tseng Huang-Chun; Ovaa Huib; Wei Nancy J C; Ploegh Hidde;

Tsai Li-Huei

CORPORATE SOURCE: Howard Hughes Medical Institute, Harvard Medical School,

Boston, Massachusetts 02155, USA.. huang-

chun tseng@hms.harvard.edu

SOURCE: Chemistry & biology, (2005 Jul) Vol. 12, No. 7, pp. 769-77.

Journal code: 9500160. ISSN: 1074-5521.

PUB. COUNTRY: England: United Kingdom

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 200510

ENTRY DATE: Entered STN: 26 Jul 2005

Last Updated on STN: 28 Oct 2005 Entered Medline: 27 Oct 2005

AB A comprehensive study of global phosphorylation events in biological systems is critical. We report a chemistry-based capture-release-tag

method for isolation of complex phospho-Ser/Thr-containing peptides by liquid beta-elimination combined with solid-phase Michael addition. The free thiol groups of 6-(mercapto-acetylamino)-hexanoic acid functionalized resin are used as immobilized Michael donors to capture dehydro-serine/threonine peptides. After an acid-mediated release step, phospho-peptides are labeled with a 6-(2-mercapto-acetylamine)-hexanoic amide tag at phosphorylated sites. We applied this method to analyze the phosphorylation status of microtubule-associated proteins. We find that a CDK5 substrate microtubule-associated protein 2 (MAP2) is phosphorylated on residues that are within a homologous region of Tau. The chemical method corroborates previous results and suggests that Tau and MAP2 may contain a CDK5 phosphorylation motif.

L14 ANSWER 3 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 2005:395356 BIOSIS DOCUMENT NUMBER: PREV200510185388

TITLE: Comparative phosphoproteomic analysis of synaptosomal

proteins of a progressive neurodegeneration mouse model. Tseng, Huang-Chun [Reprint Author]; Ovaa, Huib; Chang,

Nancy; Ploegh, Hidde; Tsai, Li-Huei

CORPORATE SOURCE: Howard Hughes Med Inst, Boston, MA 02115 USA

SOURCE: FASEB Journal, (MAY 14 2004) Vol. 18, No. 8, Suppl. S, pp.

C61.

Meeting Info.: Annual Meeting of the American-Society-for-Biochemistry-and-Molecular-Biology/8th Congress of the International-Union-for-Biochemistry-and-Molecular-Biology. Boston, MA, USA. June 12 -16, 2004. Amer Soc BioChem & Mol

Biol; Int Union Biochem & Mol Biol. CODEN: FAJOEC. ISSN: 0892-6638.

DOCUMENT TYPE: Conference; (Meeting)

Conference; Abstract; (Meeting Abstract)

LANGUAGE: English

AUTHOR(S):

ENTRY DATE: Entered STN: 5 Oct 2005

Last Updated on STN: 5 Oct 2005

Aberrent kinase activities in brain can lead to neurodegeneration. We use a novel chemistry-based capture-release-tag method for the isolation of phospho-Ser/Thr-containing peptides by beta-elimination followed by a solid-phase Michael addition. After the chemistry, phosphate at the phosphorylation sites will be replaced a specific chemical tag. Tagged serines and threonines can then be identified by tandem mass spectrometry. We analyze phosphorylation sites of synaptosomal proteins prepared from control brains and p25(Cdk5 activator) transgenic mouse brains with different degree of neurodegeneration. Results from phosphopeptide clustering analysis reveal several distinct groups of phosphorylation, suggesting changes of specific kinase/phosphase activities during neurodegeneration. Several novel hypothesis are created, based the phosphopeptide cluster data.

L14 ANSWER 4 OF 10 MEDLINE on STN DUPLICATE 3

ACCESSION NUMBER: 2003424036 MEDLINE DOCUMENT NUMBER: PubMed ID: 12964774

TITLE: Characterization of protein phosphorylation by mass

spectrometry using immobilized metal ion affinity

chromatography with on-resin

beta-elimination and Michael addition.

AUTHOR: Thompson Andrew J; Hart Sarah R; Franz Clemens; Barnouin

Karin; Ridley Anne; Cramer Rainer

CORPORATE SOURCE: The Ludwig Institute for Cancer Research, Cruciform

Building, Gower Street, London WC1E 6BT, United Kingdom.

SOURCE: Analytical chemistry, (2003 Jul 1) Vol. 75, No. 13, pp.

3232-43.

Journal code: 0370536. ISSN: 0003-2700.

PUB. COUNTRY: United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 200312

ENTRY DATE: Entered STN: 11 Sep 2003

Last Updated on STN: 18 Dec 2003 Entered Medline: 10 Dec 2003

AB A protocol combining immobilized metal ion affinity chromatography and beta-elimination with concurrent Michael addition has been developed for enhanced analysis of protein phosphorylation. Immobilized metal ion affinity chromatography was initially used to enrich for phosphorylated peptides. Beta-elimination, with or without concurrent Michael addition, was then subsequently used to simultaneously elute and derivatize phosphopeptides bound to the chromatography resin. Derivatization of the phosphate facilitated the precise determination of phosphorylation sites by MALDI-PSD/LIFT tandem mass spectrometry, avoiding complications due to ion suppression and phosphate lability in mass spectrometric analysis of phosphopeptides. Complementary use of immobilized metal ion affinity chromatography and beta-elimination with concurrent Michael addition in this manner circumvented several inherent disadvantages of the individual methods. In particular, (i) the protocol discriminated O-linked glycosylated peptides from phosphopeptides prior to betaelimination/Michael addition and (ii) the elution of peptides from the chromatography resin as derivatized phosphopeptides distinguished them from unphosphorylated species that were also retained. The chemical derivatization of phosphopeptides greatly increased the information obtained during peptide sequencing by mass spectrometry. The combined protocol enabled the detection and sequencing of phosphopeptides from protein digests at low femtomole concentrations of initial sample and was employed to identify novel phosphorylation sites on the cell adhesion protein p120 catenin and the glycoprotein fetuin.

L14 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2003:573681 CAPLUS

DOCUMENT NUMBER:

139:381153

TITLE:

Selenium-linking strategy for traceless solid-phase

synthesis of acrylamides

AUTHOR(S):

Sheng, Shou-Ri; Wang, Xing-Cong; Liu, Xiao-Ling; Song,

Cai-Sheng

CORPORATE SOURCE:

Department of Chemistry, Jiangxi Normal University,

Nanchang, Peop. Rep. China

SOURCE:

Synthetic Communications (2003), 33(16), 2867-2872

CODEN: SYNCAV; ISSN: 0039-7911

PUBLISHER:

Marcel Dekker, Inc.

DOCUMENT TYPE:

Journal English

24

LANGUAGE:
OTHER SOURCE(S):

CASREACT 139:381153

AB A novel polystyrene-supported β -selenopropionic acid was prepared and applied to simple and efficient synthesis of acrylamides. Polystyrene-supported selenium bromide was metalated by NaBH4 and reacted with 3-bromopropionic acid to give supported 3-selenylpropionic acid,

which was converted to acyl chloride and reacted with R1R2NH (R2 = H, R1 = Ph, 4-MeC6H4, 4-FC6H4, 4-ClC6H4, 4-BrC6H4, PhCH2, Bu; R1 = R2 = iPr; R1R2NH = piperidine) to give corresponding amides. The H2O2 oxidation of the amides with subsequent β -elimination gave substituted acrylamides.

REFERENCE COUNT:

THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L14 ANSWER 6 OF 10 MEDLINE on STN DUPLICATE 4

ACCESSION NUMBER: 2002444688 MEDLINE DOCUMENT NUMBER: PubMed ID: 12201785

TITLE: Versatile "traceless" sulfone linker for SPOS: preparation

of isoxazolinopyrrole 2-carboxylates.

AUTHOR:

Hwang Sung Hee; Kurth Mark J

CORPORATE SOURCE: Department of Chemistry, University of California, One

Shields Avenue, Davis, California 95616-5295, USA.

SOURCE: The Journal of organic chemistry, (2002 Sep 6) Vol. 67, No.

18, pp. 6564-7.

Journal code: 2985193R. ISSN: 0022-3263.

PUB. COUNTRY:

United States

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE:

English

FILE SEGMENT:

NONMEDLINE; PUBMED-NOT-MEDLINE

ENTRY MONTH:

200304

ENTRY DATE:

Entered STN: 31 Aug 2002

Last Updated on STN: 6 Apr 2003

Entered Medline: 4 Apr 2003

AB A five-step solid-phase synthesis of isoxazolinopyrrole-2-carboxylates (6) that employs a traceless sulfone linker strategy is reported. Resin-bound diene 4, obtained by acetylation and concomitant beta-elimination of acetate from resin-bound allylic alcohol

3, underwent regionelective 1.3-dipolar cycloadditons with pitrile oxides

3, underwent regioselective 1,3-dipolar cycloadditons with nitrile oxides. Formation of the pyrrole products in a resin-releasing strategy was performed by pyrrole annulation with alkyl isocyanoacetates, which react with the vinyl sulfone moiety to generate the target isoxazolinopyrrole-2-carboxylates (6). Use of this chemistry afforded eight

isoxazolinopyrrole-2-carboxylates in 6-24% overall yields from polystyrene/divinylbenzene sulfinate 1.

L14 ANSWER 7 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: DOCUMENT NUMBER:

2003:54648 BIOSIS PREV200300054648

TITLE:

Improved enrichment of phosphopeptides by IMAC with on-

resin beta-elimination and

Michael addition.

AUTHOR(S):

Thompson, Andrew; Hart, Sarah; Barnouin, Karin; Cramer,

Rainer

SOURCE:

Molecular & Cellular Proteomics, (September 2002) Vol. 1,

No. 9, pp. 698. print.

Meeting Info.: First World Congress of the Human Proteome Organisation. Versailles, Paris, France. November 21-24,

2002. Human Proteome Organisation.

ISSN: 1535-9476 (ISSN print).

DOCUMENT TYPE:

Conference; (Meeting)

Conference; Abstract; (Meeting Abstract)

LANGUAGE:

English

ENTRY DATE:

Entered STN: 22 Jan 2003

Last Updated on STN: 22 Jan 2003

L14 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2001:640874 CAPLUS

TITLE:

Traceless sulfone linker in solid phase synthesis: Preparation of 3,5-disubstituted cyclopent-2-enone

AUTHOR(S):

Kurth, Mark J.; Cheng, Wei-Chieh

CORPORATE SOURCE:

Department of Chemistry, University of California,

SOURCE:

Davis, Davis, CA, 95616, USA

Abstracts of Papers, 222nd ACS National Meeting, Chicago, IL, United States, August 26-30, 2001 (2001), ORGN-511. American Chemical Society: Washington, D.

С.

CODEN: 69BUZP

DOCUMENT TYPE:

Conference; Meeting Abstract

LANGUAGE: English

AB The preparation of functionalized 3,5-disubstituted cyclopent-2-enone via a traceless solid-phase sulfone linker strategy is described.

Polystyrene/divinylbenzene sulfinate 1 undergoes S-alkylation followed by

 α, α -dialkyltion with 1,4-dichloro-2-butene to form

polymer-bound 3-phenylsulfonylcyclopentene species. Subsequent epoxidn.

was accomplished by treating mCPBA. Oxirane moiety of resin 11 was opened with various nucleophiles such as Gringerd regent, higher order Cuprate reagent, azide ion, and amines. To complete a traceless linker cleavage strategy, swern oxidation was employed to generate polymer-bound 3-phenylsulfonylcyclopentanes and simultaneous release 3,5-disubstituted cyclopent-2-enones from the resin via .beta.elimination of the carbonyl moiety. Twelve 3,5-disubstituted cyclopent-2-enones were prepared in five steps and 20-42% overall yield from polystyrene/divinylbenzene sulfinate 1.

L14 ANSWER 9 OF 10 MEDLINE on STN DUPLICATE 5

ACCESSION NUMBER: 96016037 MEDLINE DOCUMENT NUMBER: PubMed ID: 7546982

TITLE: Synthesis of an N-methyldehydroalanine-containing fragment

of microcystin by combination of solid

phase peptide synthesis and beta-

elimination in solution.

AUTHOR: Zetterstrom M; Trogen L; Hammarstrom L G; Juhlin L; Nilsson

B; Damberg C; Bartfai T; Langel U

CORPORATE SOURCE: Department of Neurochemistry and Neurotoxicology, Arrhenius

Laboratories, Stockholm University, Sweden.

SOURCE: Acta chemica Scandinavica (Copenhagen, Denmark: 1989),

> (1995 Sep) Vol. 49, No. 9, pp. 696-700. Journal code: 9012772. ISSN: 0904-213X.

PUB. COUNTRY: Denmark

DOCUMENT TYPE: Journal; Article; (JOURNAL ARTICLE)

LANGUAGE: English

FILE SEGMENT: Priority Journals

ENTRY MONTH: 199510

ENTRY DATE: Entered STN: 27 Dec 1995

> Last Updated on STN: 27 Dec 1995 Entered Medline: 27 Oct 1995

AB A new method for the synthesis of dehydroalanine (delta Ala)-containing peptides has been developed by combining solid phase peptide synthesis (tert-butyloxycarbonyl/HF-chemistry) with solution synthesis. A sequence from cyanobacterial hepatotoxin microcystin, Ac-D-gamma-Glu-[N-Me-delta Ala]-D-Ala-Leu amide was chosen as a model peptide. The precursor for the synthesis of the dehydroalanine-containing peptide, Ac-D-gamma-Glu-[N,SdiMeCys]-D-Ala-Leu, was synthesized on a solid phase followed by sulfonium salt formation on the resin. The resulting S,S-dimethylated peptide was cleaved from the resin with liquid HF. The HPLC-purified S,S-dimethylated cysteine-containing precursor peptide was subjected to beta-elimination in solution catalysed by DBU (1,8-diazabicyclo[5.4.0]undec-7-ene) in methanol. The final product, Ac-D-gamma-Glu-[N-Me-delta Ala]-D-Ala-Leu amide, was purified by HPLC, and analysed by mass spectrometry and 1H NMR spectroscopy. The stability of the model peptide under acidic, neutral and basic conditions has been studied.

L14 ANSWER 10 OF 10 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 1991:450915 BIOSIS

DOCUMENT NUMBER: PREV199192095695; BA92:95695

TITLE: CHEMOSELECTIVE ONE-STEP PURIFICATION METHOD FOR PEPTIDES

SYNTHESIZED BY THE SOLID-PHASE TECHNIQUE.

AUTHOR(S):FUNAKOSHI S [Reprint author]; FUKUDA H; FUJII N

CORPORATE SOURCE: FAC PHARMACEUTICAL SCI, KYOTO UNIV, SAKYO-KU, KYOTO 606,

SOURCE: Proceedings of the National Academy of Sciences of the

United States of America, (1991) Vol. 88, No. 16, pp.

6981-6985.

CODEN: PNASA6. ISSN: 0027-8424.

DOCUMENT TYPE: Article

FILE SEGMENT: BA LANGUAGE: **ENGLISH** ENTRY DATE:

Entered STN: 11 Oct 1991 Last Updated on STN: 11 Oct 1991

The specific reaction between SH and iodoacetamide groups has been explored as the basis of an affinity-type purification procedure for peptides synthesized by the solid-phase technique. For this affinity-type purification procedure, we synthesized an SH precursor reagent bearing an acid-labile S-protecting group, pMB-SCH2CONHCH2CH2SO2CH2CH2OCO2pNP (compound I), in which pMB is p-methoxybenzyl and pNP is p-nitrophenyl. Using this reagent, the procedure involves the following sequence of four reactions: (i) attachment of the SH function of compound I to the α -amino group of a peptide-resin through a base-labile sulfonylethoxycarbonyl linkage in the final step of solid-phase peptide synthesis, (ii) acid treatment to remove the S-pMB and side-chain-protecting groups employed and cleave the modified peptide from the resin, (iii) immobilization of the derived SH-peptide on an iodoacetamide-resin column, and (iv) base (5% NH4OH) treatment to release the desired peptide from the resin in nearly pure form. To facilitate this purification procedure, unreacted amino groups were acetylated in each step during solid-phase synthesis. The usefulness of this method was demonstrated by the purification of several peptides (18 to \approx 44 amino acids in length) synthesized by the 9-fluorenylmethoxycarbonyl (Fmoc)-based solid-phase technique. The principle of this affinity-type purification procedure may also be applied to the tert-butoxycarbonyl (Boc)-based solid-phase technique.